



## Structure of $\Lambda(\delta\lambda\lambda)$ -[Co(en)<sub>3</sub>]I<sub>3</sub>(I)<sub>2</sub>

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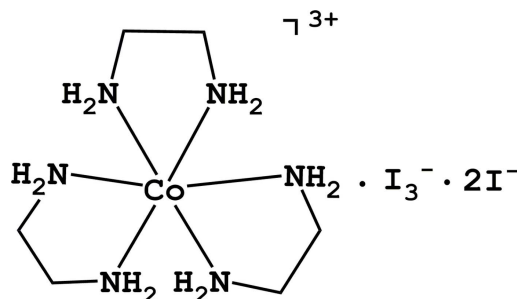
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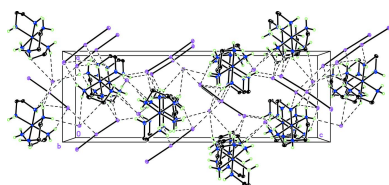
The structure of tris(ethane-1,2-diamine- $\kappa^2N,N'$ )cobalt(III) bis(iodide) triiodide, [Co(C<sub>2</sub>H<sub>8</sub>N<sub>2</sub>)<sub>3</sub>]I<sub>3</sub>(I)<sub>2</sub>, at 120 K has orthorhombic (*P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>) symmetry. The diamine nitrogen atoms form N—H...I hydrogen bonds throughout the lattice, resulting in a three-dimensional network, which involves the iodide and all atoms in the triiodide anions.

### 1. Chemical context

Significant information on the hydrogen bonding and other interactions that contribute to the chiral discriminations between metal-ion complexes has been obtained from the crystal structures of compounds containing a chiral complex cation and a chiral complex anion (Warren *et al.*, 1994; Marusak & Lappin, 1989). For example, a comparison of the compounds  $\Lambda$ -[Co(en)<sub>3</sub>] $\Delta$ -[Co(en)(ox)<sub>2</sub>]I<sub>2</sub>·3H<sub>2</sub>O and  $\Delta$ -[Co(en)<sub>3</sub>] $\Delta$ -[Co(en)(ox)<sub>2</sub>]I<sub>2</sub>·H<sub>2</sub>O reveals the importance of different helicities projected along the C<sub>3</sub> and C<sub>2</sub> axes of [Co(en)<sub>3</sub>]<sup>3+</sup> in discriminating with the pseudo-C<sub>3</sub> face of the  $\Delta$ -[Co(en)(ox)<sub>2</sub>]<sup>−</sup> anion (Lappin *et al.*, 1993).



As part of a study involving potential effects of non-chiral counter-ions, an attempt was made to grow crystals with [Co(en)<sub>3</sub>]I<sub>3</sub> and Na[Co(edta)]. However, in the presence of I<sup>−</sup>, the mildly oxidizing [Co(edta)]<sup>−</sup> was reduced and an unexpected product, [Co(en)<sub>3</sub>]I<sub>3</sub>(I)<sub>2</sub> was obtained. The structure of the corresponding cobalt(II) complex, [Co(en)<sub>3</sub>]I<sub>3</sub>I, has been reported (Du *et al.*, 2007). The larger cobalt(II) complex supports an *lel*<sub>3</sub> geometry of the bidentate ligands around the cobalt center. The Co—N bond distances in [Co(en)<sub>3</sub>]<sup>2+</sup> average 2.28 Å, significantly longer than the 1.97 Å average in [Co(en)<sub>3</sub>]<sup>3+</sup> and consistent with the sluggish redox exchange between the complexes (Jolley *et al.*, 1990). In [Co(en)<sub>3</sub>]I<sub>3</sub>I, the I<sup>−</sup> ions are located along the quasi-C<sub>2</sub> axis of the [Co(en)<sub>3</sub>]<sup>2+</sup> complex ion with close hydrogen-bond contacts from N—H protons of 2.91 Å. The terminal iodine atoms of the I<sub>3</sub><sup>−</sup> ions likewise form hydrogen bonds with N—H protons at 2.93 Å, resulting in an alternating chain of linear I<sub>3</sub><sup>−</sup> ions at 90° to one another down the *c*-axis direction.

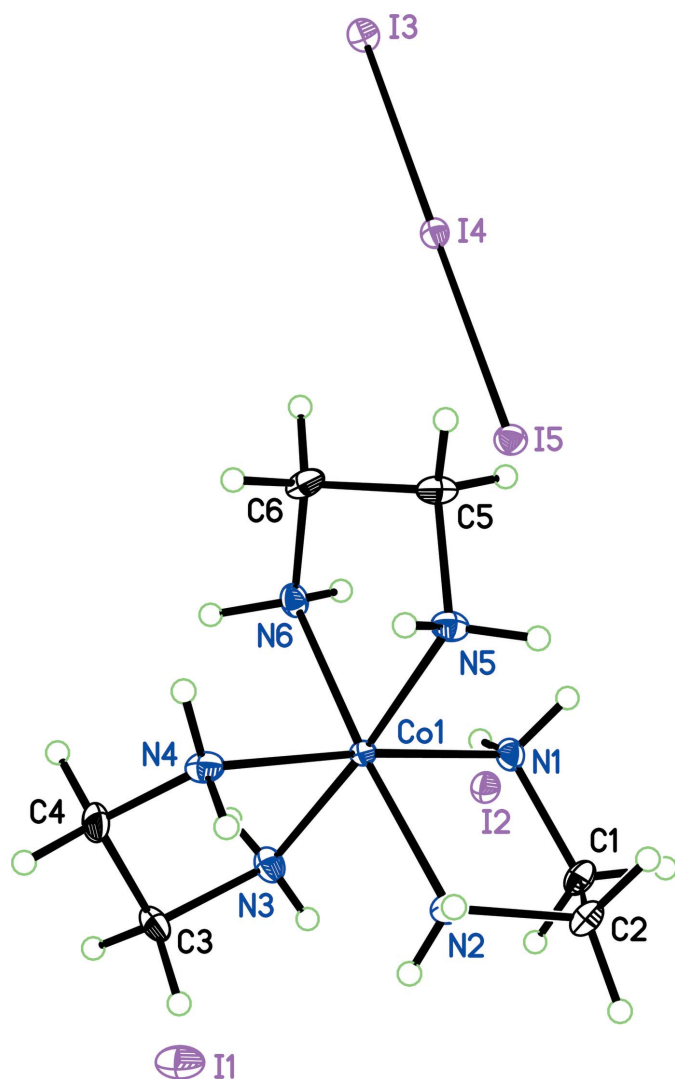


**Table 1**  
 Selected geometric parameters (Å, °).

Co1—N1	1.964 (3)	Co1—N3	1.968 (3)
Co1—N5	1.967 (3)	Co1—N6	1.969 (3)
Co1—N4	1.968 (3)	Co1—N2	1.982 (3)
N1—Co1—N5	91.86 (14)	N4—Co1—N6	91.39 (13)
N1—Co1—N4	175.38 (14)	N3—Co1—N6	91.83 (13)
N5—Co1—N4	91.64 (14)	N1—Co1—N2	85.20 (13)
N1—Co1—N3	91.25 (13)	N5—Co1—N2	91.96 (14)
N5—Co1—N3	175.65 (14)	N4—Co1—N2	91.68 (13)
N4—Co1—N3	85.42 (14)	N3—Co1—N2	91.34 (15)
N1—Co1—N6	91.91 (13)	N6—Co1—N2	175.76 (13)
N5—Co1—N6	85.02 (13)		

## 2. Structural commentary

The complex,  $[\text{Co}(\text{en})_3](\text{I}_3)(\text{I})_2$  crystallizes as dark-red, rod-like crystals. The asymmetric unit of the primitive, acentric, orthorhombic space group  $P2_12_12_1$  consists of one  $[\text{Co}(\text{en})_3]^{3+}$  cation, two iodide anions and a triiodide anion (Fig. 1). The correct enantiomorph of the space group was determined by



**Figure 1**  
 Labeling scheme for  $[\text{Co}(\text{en})_3]\text{I}_3(\text{I})_2$  with ellipsoids at the 50% probability level. Hydrogen atoms depicted as spheres of an arbitrary radius.

**Table 2**  
 Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1C $\cdots$ I2	0.87 (5)	2.83 (5)	3.598 (3)	149 (4)
N1—H1D $\cdots$ I2 <sup>i</sup>	0.87 (5)	2.79 (5)	3.616 (3)	158 (4)
N2—H2C $\cdots$ I3 <sup>iii</sup>	0.89 (5)	3.04 (5)	3.823 (3)	149 (4)
N2—H2D $\cdots$ I1 <sup>iii</sup>	0.80 (5)	3.02 (5)	3.756 (4)	154 (4)
N3—H3C $\cdots$ I2 <sup>iv</sup>	0.97 (5)	3.25 (5)	4.111 (3)	149 (4)
N3—H3C $\cdots$ I5 <sup>iv</sup>	0.97 (5)	3.08 (5)	3.586 (3)	114 (3)
N3—H3D $\cdots$ I1 <sup>iii</sup>	0.76 (5)	3.18 (5)	3.805 (4)	142 (5)
N4—H4C $\cdots$ I1	0.92 (5)	2.77 (5)	3.630 (3)	155 (4)
N4—H4D $\cdots$ I1 <sup>v</sup>	0.90 (5)	2.90 (5)	3.715 (3)	152 (4)
N5—H5C $\cdots$ I1 <sup>v</sup>	0.89 (5)	2.95 (5)	3.765 (3)	154 (4)
N5—H5C $\cdots$ I3 <sup>iii</sup>	0.89 (5)	3.25 (5)	3.639 (3)	109 (3)
N5—H5D $\cdots$ I4 <sup>ii</sup>	0.89 (5)	3.05 (5)	3.611 (3)	123 (4)
N6—H6C $\cdots$ I5	0.91 (5)	2.89 (5)	3.674 (3)	145 (4)
N6—H6D $\cdots$ I2 <sup>iv</sup>	0.95 (5)	2.80 (5)	3.684 (3)	157 (4)

Symmetry codes: (i)  $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$ ; (ii)  $-x, y+\frac{1}{2}, -z+\frac{1}{2}$ ; (iii)  $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$ ; (iv)  $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$ ; (v)  $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$ .

comparison of intensities of Friedel pairs of reflections, yielding a Flack  $x$  parameter of 0.017 (9) (Parsons *et al.*, 2013) and a Hooft  $y$  parameter of 0.006 (8) (Hooft *et al.*, 2008). Values close to zero indicate the correct enantiomorph of the space group. This determination allows an accurate assessment of the configuration of the cobalt cation.

The cobalt center is located in a slightly distorted octahedral environment by the nitrogen atoms of three ethylene diamine ligands (see Table 1 for details). The ligands adopt a  $\Lambda(\delta\lambda\lambda)$  *lel ob ob* (*lelob*<sub>2</sub>) geometry about the cobalt center, Fig. 1. Bond distances and angles within the molecules are unexceptional.

The amine hydrogen atoms were initially located from a difference-Fourier map and were refined freely. All of the amine hydrogen atoms are involved in hydrogen bonds to nearby iodine/triiodide moieties, Fig. 2. This interconnectivity results in a three-dimensional hydrogen-bonded network throughout the entire structure.

## 3. Supramolecular features

The iodide ion  $\text{I}^-$ (1) is hydrogen bonded to N—H protons from N4 on one  $[\text{Co}(\text{en})_3]^{3+}$  ion at 2.77 Å, bridging to N—H protons on N4 and N5 from the two ligands with a  $\lambda$ -configuration on an adjacent cation with distances of 2.90 (5) and 2.95 (5) Å (Fig. 2, Table 2). The pairwise interactions create a hydrogen-bonded chain along the crystallographic  $a$ -axis direction, forming a layer with the complex cations separated by channels formed by  $\text{I}_3^-$  ions in an alternating herringbone pattern punctuated by  $\text{I}_2^-$  ions. The iodide  $\text{I}_2^-$  forms a hydrogen-bonded network bridging the layers with N—H protons from three separate cations at 2.79 (5), 2.80 (5) and 2.83 (5) Å. The  $\text{I}_3^-$  ion has a close N—H contact with N6 at 2.89 (5) Å.

## 4. Database survey

A survey of  $\text{Co}(\text{en})_3$  coupled with iodine reveals 23 structures in the Cambridge Structural Database (CSD v5.42, November

2020; Groom *et al.*, 2016). Predominantly these are Co(III) complexes. There are three reports of  $\text{Co}(\text{en})_3\text{I}_3$  (EDANEC, Matsuki *et al.*, 2001; ENCOIH, Whuler *et al.*, 1980; FIXLAI, Grant *et al.*, 2019). EDANEC and FIXLAI are structural analyses of the  $\Lambda$ - and  $\Delta$ -isomers, respectively. The structure determination by Whuler *et al.* is of the racemic cation species. A mixed Cl/I species was reported by Huang and co-workers (FAXMEX, Zhang *et al.*, 2005). All of these reports also contain water of crystallization. There is one report of  $\text{Co}(\text{en})_3$  that has both an iodide and a triiodide pair of counter-ions that crystallizes in the tetragonal space group  $I\bar{4}2d$  (HIQYUC, Du *et al.*, 2007). However, that report is of the  $\text{Co}^{\text{II}}$  complex,  $\text{Co}(\text{en})_3(\text{I}_3)\text{I}$ .

### 5. Synthesis and crystallization

Crystals were obtained from an attempt to co-crystallize optically active  $[\text{Co}(\text{en})_3]^{3+}$  and the mildly oxidizing  $[\text{Co}(\text{edta})]^-$  from  $\Lambda$ - $[\text{Co}(\text{en})_3]\text{I}_3$  and  $\text{Na}[\text{Co}(\text{edta})]$ . After storage at 283 K for two weeks, the deep-purple coloration of the  $[\text{Co}(\text{edta})]^-$  ion had disappeared and dark-red well-formed crystals were recovered.

### 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The structure was solved by dual-space methods (Sheldrick, 2015a) and refined routinely (Sheldrick, 2015b). Amine hydrogen atoms were refined freely and methylene hydrogen atoms were refined as riding on the carbon to which they are bonded with  $\text{C}-\text{H} = 0.99 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

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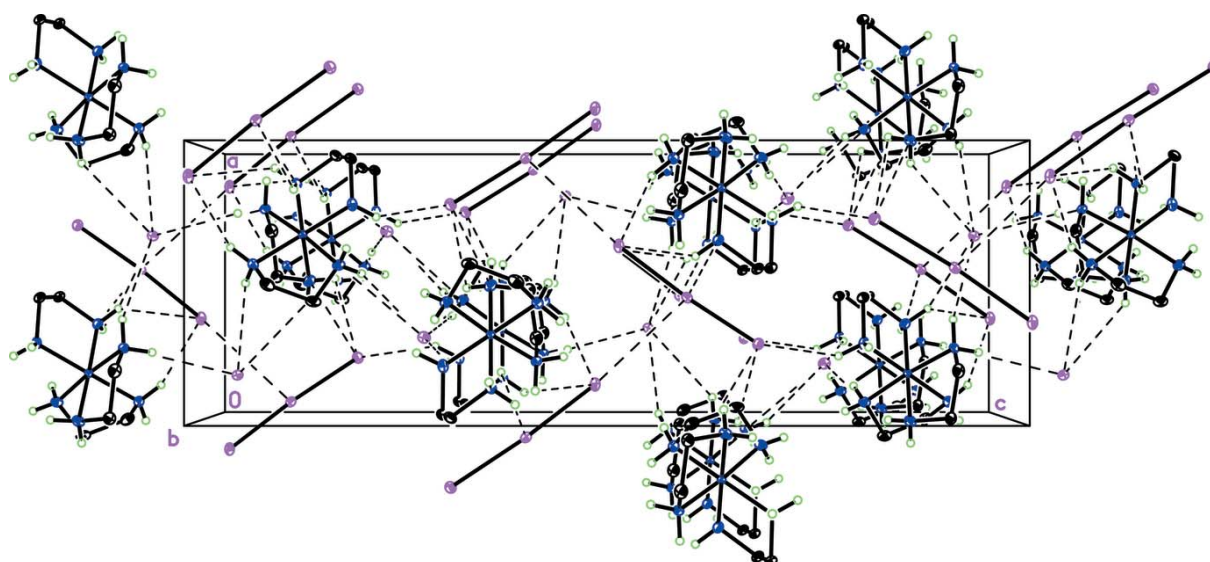
**Table 3**  
Experimental details.

Crystal data	
Chemical formula	$[\text{Co}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{I}_3(\text{I})_2$
$M_r$	873.74
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	120
$a, b, c$ (Å)	8.7508 (12), 8.8333 (12), 25.982 (4)
$V$ (Å <sup>3</sup> )	2008.4 (5)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	8.54
Crystal size (mm)	0.28 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker Kappa X8 APEXII
Absorption correction	Numerical ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015)
$T_{\text{min}}, T_{\text{max}}$	0.603, 1.000
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	35617, 5024, 5023
$R_{\text{int}}$	0.022
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.012, 0.028, 1.33
No. of reflections	5024
No. of parameters	199
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.39, -0.66
Absolute structure	Flack $x$ determined using 2136 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013).
Absolute structure parameter	0.017 (9)

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2020), *CIFTAB* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

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**Figure 2**  
View down the  $b$  axis of  $[\text{Co}(\text{en})_3]\text{I}_3(\text{I})_2$  showing the herringbone pattern, with ellipsoids at the 50% probability level.

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## supporting information

*Acta Cryst.* (2021). E77, 446-449 [https://doi.org/10.1107/S2056989021002826]

Structure of  $\Lambda(\delta\lambda\lambda)$ -[Co(en)<sub>3</sub>]I<sub>3</sub>(I)<sub>2</sub>

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## Computing details

Data collection: *APEX3* (Bruker, 2015); cell refinement: *SAINT* (Bruker, 2015); data reduction: *SAINT* (Bruker, 2015); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *CIFTAB* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Tris(ethane-1,2-diamine- $\kappa^2N,N'$ )cobalt(III) bis(iodide) triiodide

## Crystal data

[Co(C<sub>2</sub>H<sub>8</sub>N<sub>2</sub>)<sub>3</sub>]I<sub>3</sub>(I)<sub>2</sub>

$M_r = 873.74$

Orthorhombic,  $P2_12_12_1$

$a = 8.7508$  (12) Å

$b = 8.8333$  (12) Å

$c = 25.982$  (4) Å

$V = 2008.4$  (5) Å<sup>3</sup>

$Z = 4$

$F(000) = 1576$

$D_x = 2.890$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9680 reflections

$\theta = 2.4$ – $28.4^\circ$

$\mu = 8.54$  mm<sup>-1</sup>

$T = 120$  K

Rod, dark red

$0.28 \times 0.15 \times 0.10$  mm

## Data collection

Bruker Kappa X8 APEXII  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.33 pixels mm<sup>-1</sup>

combination of  $\omega$  and  $\varphi$ -scans

Absorption correction: numerical  
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.603$ ,  $T_{\max} = 1.000$

35617 measured reflections

5024 independent reflections

5023 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 1.6^\circ$

$h = -11 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -33 \rightarrow 34$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.012$

$wR(F^2) = 0.028$

$S = 1.33$

5024 reflections

199 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier  
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0073P)^2 + 1.9769P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.39$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.66$  e Å<sup>-3</sup>

Absolute structure: Flack  $x$  determined using  
2136 quotients  $[(I^+) - (I^-)] / [(I^+) + (I^-)]$  (Parsons *et al.*, 2013)

Absolute structure parameter: 0.017 (9)

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.32971 (3)	0.22384 (3)	0.55282 (2)	0.01429 (5)
I2	0.69407 (3)	0.30959 (2)	0.22059 (2)	0.01325 (5)
I3	−0.13267 (3)	0.17300 (3)	0.01437 (2)	0.01367 (5)
I4	0.05242 (3)	0.28734 (2)	0.09775 (2)	0.01107 (5)
I5	0.22245 (3)	0.40722 (3)	0.18675 (2)	0.01307 (5)
Co1	0.32630 (5)	0.30665 (5)	0.36719 (2)	0.00693 (8)
N1	0.4364 (4)	0.4391 (3)	0.31911 (13)	0.0111 (6)
H1C	0.466 (6)	0.383 (5)	0.2936 (18)	0.013*
H1D	0.380 (6)	0.513 (5)	0.3077 (18)	0.013*
N2	0.4337 (4)	0.4219 (4)	0.42174 (12)	0.0119 (6)
H2C	0.374 (6)	0.453 (5)	0.4473 (19)	0.014*
H2D	0.500 (6)	0.370 (5)	0.4332 (19)	0.014*
N3	0.4917 (4)	0.1557 (3)	0.36466 (13)	0.0121 (6)
H3C	0.484 (6)	0.087 (5)	0.3358 (18)	0.015*
H3D	0.571 (6)	0.189 (6)	0.3664 (19)	0.015*
N4	0.2318 (4)	0.1713 (4)	0.41825 (12)	0.0122 (6)
H4C	0.248 (5)	0.217 (5)	0.4497 (19)	0.015*
H4D	0.129 (6)	0.168 (5)	0.4168 (18)	0.015*
N5	0.1500 (4)	0.4446 (3)	0.36922 (12)	0.0114 (6)
H5C	0.094 (6)	0.419 (5)	0.3963 (19)	0.014*
H5D	0.173 (6)	0.543 (5)	0.3687 (18)	0.014*
N6	0.2158 (4)	0.2066 (3)	0.31060 (12)	0.0109 (5)
H6C	0.261 (5)	0.236 (5)	0.2807 (19)	0.013*
H6D	0.227 (5)	0.100 (5)	0.3128 (18)	0.013*
C1	0.5641 (4)	0.5172 (4)	0.34653 (16)	0.0154 (7)
H1A	0.595861	0.608878	0.327383	0.018*
H1B	0.653317	0.448913	0.349678	0.018*
C2	0.5050 (5)	0.5596 (4)	0.39899 (16)	0.0158 (7)
H2A	0.589927	0.595429	0.421034	0.019*
H2B	0.428560	0.641804	0.396078	0.019*
C3	0.4761 (4)	0.0485 (4)	0.40885 (15)	0.0129 (7)
H3A	0.521899	0.092664	0.440276	0.016*
H3B	0.528980	−0.047957	0.401093	0.016*
C4	0.3078 (5)	0.0210 (4)	0.41678 (15)	0.0146 (7)
H4A	0.266124	−0.040706	0.388242	0.018*
H4B	0.290451	−0.033783	0.449508	0.018*
C5	0.0500 (4)	0.4213 (4)	0.32313 (15)	0.0135 (7)
H5A	0.089273	0.479960	0.293487	0.016*
H5B	−0.055557	0.455093	0.330595	0.016*

C6	0.0521 (4)	0.2540 (4)	0.31111 (15)	0.0135 (7)
H6A	-0.005054	0.196883	0.337647	0.016*
H6B	0.004415	0.234598	0.277189	0.016*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
I1	0.00933 (10)	0.02215 (11)	0.01140 (10)	0.00063 (9)	-0.00073 (9)	-0.00018 (9)
I2	0.01308 (11)	0.01112 (9)	0.01555 (11)	-0.00011 (8)	-0.00068 (9)	-0.00056 (8)
I3	0.01745 (12)	0.01416 (10)	0.00939 (10)	0.00078 (8)	0.00012 (9)	-0.00136 (8)
I4	0.01061 (10)	0.01122 (9)	0.01138 (10)	0.00010 (8)	0.00211 (8)	0.00068 (8)
I5	0.01294 (11)	0.01450 (10)	0.01176 (11)	-0.00060 (8)	-0.00106 (9)	0.00081 (8)
Co1	0.0067 (2)	0.00784 (19)	0.00628 (19)	-0.00032 (16)	0.00029 (17)	0.00003 (15)
N1	0.0095 (14)	0.0099 (13)	0.0139 (15)	-0.0003 (11)	0.0011 (13)	0.0024 (11)
N2	0.0109 (15)	0.0144 (14)	0.0104 (14)	-0.0011 (12)	-0.0008 (12)	-0.0011 (11)
N3	0.0116 (15)	0.0110 (13)	0.0138 (15)	0.0019 (11)	0.0024 (13)	0.0024 (11)
N4	0.0083 (14)	0.0189 (14)	0.0095 (14)	-0.0023 (12)	0.0009 (12)	0.0023 (12)
N5	0.0108 (15)	0.0130 (14)	0.0104 (14)	0.0028 (11)	-0.0015 (12)	-0.0024 (11)
N6	0.0156 (14)	0.0080 (12)	0.0091 (13)	0.0005 (11)	-0.0008 (12)	-0.0004 (10)
C1	0.0100 (17)	0.0149 (16)	0.0212 (19)	-0.0073 (14)	-0.0008 (16)	0.0043 (14)
C2	0.0150 (18)	0.0125 (16)	0.020 (2)	-0.0049 (14)	-0.0044 (16)	-0.0013 (14)
C3	0.0147 (18)	0.0117 (15)	0.0124 (17)	0.0021 (13)	0.0000 (14)	0.0048 (13)
C4	0.0187 (19)	0.0112 (14)	0.0140 (17)	-0.0041 (14)	-0.0003 (15)	0.0048 (12)
C5	0.0103 (16)	0.0175 (16)	0.0127 (17)	0.0016 (13)	-0.0033 (14)	-0.0019 (13)
C6	0.0100 (16)	0.0178 (16)	0.0126 (16)	-0.0042 (13)	-0.0018 (14)	-0.0011 (13)

*Geometric parameters (Å, °)*

I3—I4	2.8875 (4)	N5—H5C	0.89 (5)
I4—I5	2.9464 (4)	N5—H5D	0.89 (5)
Co1—N1	1.964 (3)	N6—C6	1.493 (5)
Co1—N5	1.967 (3)	N6—H6C	0.91 (5)
Co1—N4	1.968 (3)	N6—H6D	0.95 (5)
Co1—N3	1.968 (3)	C1—C2	1.505 (6)
Co1—N6	1.969 (3)	C1—H1A	0.9900
Co1—N2	1.982 (3)	C1—H1B	0.9900
N1—C1	1.494 (5)	C2—H2A	0.9900
N1—H1C	0.87 (5)	C2—H2B	0.9900
N1—H1D	0.87 (5)	C3—C4	1.507 (5)
N2—C2	1.490 (5)	C3—H3A	0.9900
N2—H2C	0.89 (5)	C3—H3B	0.9900
N2—H2D	0.80 (5)	C4—H4A	0.9900
N3—C3	1.495 (5)	C4—H4B	0.9900
N3—H3C	0.97 (5)	C5—C6	1.511 (5)
N3—H3D	0.76 (5)	C5—H5A	0.9900
N4—C4	1.486 (5)	C5—H5B	0.9900
N4—H4C	0.92 (5)	C6—H6A	0.9900
N4—H4D	0.90 (5)	C6—H6B	0.9900

N5—C5	1.498 (5)		
I3—I4—I5	176.193 (11)	Co1—N5—H5D	115 (3)
N1—Co1—N5	91.86 (14)	H5C—N5—H5D	113 (4)
N1—Co1—N4	175.38 (14)	C6—N6—Co1	109.8 (2)
N5—Co1—N4	91.64 (14)	C6—N6—H6C	110 (3)
N1—Co1—N3	91.25 (13)	Co1—N6—H6C	107 (3)
N5—Co1—N3	175.65 (14)	C6—N6—H6D	112 (3)
N4—Co1—N3	85.42 (14)	Co1—N6—H6D	111 (3)
N1—Co1—N6	91.91 (13)	H6C—N6—H6D	107 (4)
N5—Co1—N6	85.02 (13)	N1—C1—C2	106.8 (3)
N4—Co1—N6	91.39 (13)	N1—C1—H1A	110.4
N3—Co1—N6	91.83 (13)	C2—C1—H1A	110.4
N1—Co1—N2	85.20 (13)	N1—C1—H1B	110.4
N5—Co1—N2	91.96 (14)	C2—C1—H1B	110.4
N4—Co1—N2	91.68 (13)	H1A—C1—H1B	108.6
N3—Co1—N2	91.34 (15)	N2—C2—C1	107.5 (3)
N6—Co1—N2	175.76 (13)	N2—C2—H2A	110.2
C1—N1—Co1	109.8 (2)	C1—C2—H2A	110.2
C1—N1—H1C	114 (3)	N2—C2—H2B	110.2
Co1—N1—H1C	107 (3)	C1—C2—H2B	110.2
C1—N1—H1D	104 (3)	H2A—C2—H2B	108.5
Co1—N1—H1D	113 (3)	N3—C3—C4	107.2 (3)
H1C—N1—H1D	110 (4)	N3—C3—H3A	110.3
C2—N2—Co1	109.5 (2)	C4—C3—H3A	110.3
C2—N2—H2C	107 (3)	N3—C3—H3B	110.3
Co1—N2—H2C	114 (3)	C4—C3—H3B	110.3
C2—N2—H2D	108 (4)	H3A—C3—H3B	108.5
Co1—N2—H2D	108 (3)	N4—C4—C3	107.3 (3)
H2C—N2—H2D	109 (5)	N4—C4—H4A	110.3
C3—N3—Co1	109.7 (2)	C3—C4—H4A	110.3
C3—N3—H3C	101 (3)	N4—C4—H4B	110.3
Co1—N3—H3C	113 (3)	C3—C4—H4B	110.3
C3—N3—H3D	106 (4)	H4A—C4—H4B	108.5
Co1—N3—H3D	115 (4)	N5—C5—C6	107.0 (3)
H3C—N3—H3D	111 (5)	N5—C5—H5A	110.3
C4—N4—Co1	109.7 (2)	C6—C5—H5A	110.3
C4—N4—H4C	110 (3)	N5—C5—H5B	110.3
Co1—N4—H4C	106 (3)	C6—C5—H5B	110.3
C4—N4—H4D	114 (3)	H5A—C5—H5B	108.6
Co1—N4—H4D	114 (3)	N6—C6—C5	106.7 (3)
H4C—N4—H4D	102 (4)	N6—C6—H6A	110.4
C5—N5—Co1	110.6 (2)	C5—C6—H6A	110.4
C5—N5—H5C	106 (3)	N6—C6—H6B	110.4
Co1—N5—H5C	107 (3)	C5—C6—H6B	110.4
C5—N5—H5D	105 (3)	H6A—C6—H6B	108.6
Co1—N1—C1—C2	-39.6 (3)	N3—C3—C4—N4	-49.3 (4)



Co1—N2—C2—C1	-37.5 (4)	Co1—N5—C5—C6	36.0 (3)
N1—C1—C2—N2	49.9 (4)	Co1—N6—C6—C5	40.5 (3)
Co1—N3—C3—C4	37.5 (3)	N5—C5—C6—N6	-49.1 (4)
Co1—N4—C4—C3	38.7 (3)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1C...I2	0.87 (5)	2.83 (5)	3.598 (3)	149 (4)
N1—H1D...I2 <sup>i</sup>	0.87 (5)	2.79 (5)	3.616 (3)	158 (4)
N2—H2C...I3 <sup>ii</sup>	0.89 (5)	3.04 (5)	3.823 (3)	149 (4)
N2—H2D...I1 <sup>iii</sup>	0.80 (5)	3.02 (5)	3.756 (4)	154 (4)
N3—H3C...I2 <sup>iv</sup>	0.97 (5)	3.25 (5)	4.111 (3)	149 (4)
N3—H3C...I5 <sup>iv</sup>	0.97 (5)	3.08 (5)	3.586 (3)	114 (3)
N3—H3D...I1 <sup>iii</sup>	0.76 (5)	3.18 (5)	3.805 (4)	142 (5)
N4—H4C...I1	0.92 (5)	2.77 (5)	3.630 (3)	155 (4)
N4—H4D...I1 <sup>v</sup>	0.90 (5)	2.90 (5)	3.715 (3)	152 (4)
N5—H5C...I1 <sup>v</sup>	0.89 (5)	2.95 (5)	3.765 (3)	154 (4)
N5—H5C...I3 <sup>ii</sup>	0.89 (5)	3.25 (5)	3.639 (3)	109 (3)
N5—H5D...I4 <sup>ii</sup>	0.89 (5)	3.05 (5)	3.611 (3)	123 (4)
N6—H6C...I5	0.91 (5)	2.89 (5)	3.674 (3)	145 (4)
N6—H6D...I2 <sup>iv</sup>	0.95 (5)	2.80 (5)	3.684 (3)	157 (4)

Symmetry codes: (i)  $-x+1, y+1/2, -z+1/2$ ; (ii)  $-x, y+1/2, -z+1/2$ ; (iii)  $x+1/2, -y+1/2, -z+1$ ; (iv)  $-x+1, y-1/2, -z+1/2$ ; (v)  $x-1/2, -y+1/2, -z+1$ .